CLEANING PROCEDURES 3.2

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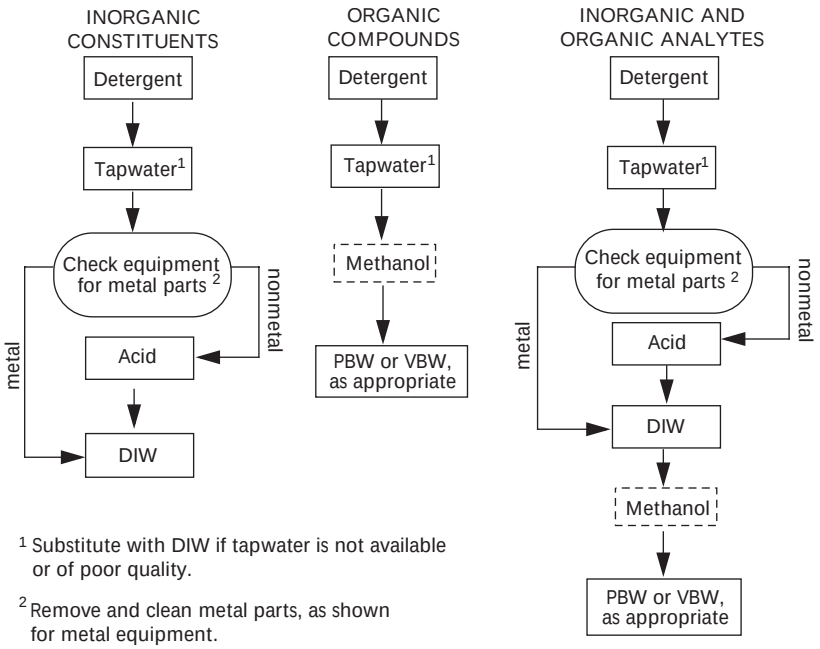
Equipment should be cleaned in an area protected from airborne or other sources of contamination. Procedures to remove contaminants to concentrations below the targeted method-detection levels can vary, depending on the cleaning supplies used, the type of equipment being cleaned, the solubility and concentration of contaminant(s), and the length of time equipment is exposed to contaminant(s). Examine equipment-blank and field-blank data to determine whether adjustments to the cleaning protocol are needed (section 3.4).

The cleaning procedure to be used depends on the type(s) of water samples that will be collected and processed. Figure 3-1 summarizes the sequence of cleaning procedures for equipment used to collect samples for inorganic and (or) organic analytes (Sandstrom, 1990; Horowitz and others, 1994; and Koterba and others, 1995).

- Inspect equipment for stains, cuts, or abrasions. Replace parts as needed.
  - Replace chipped or cracked glassware.
  - Replace bent sampler nozzles or samplers with bent fins (surface-water samplers).
  - Replace tubing if mold, mildew, or imbedded sediment cannot be removed.
  - Replace cracked or severely crimped O-rings.
  - Repair pump intakes and antibacksiphons that have loose or missing screws.
  - Check the flow manifold and sample tubing to ensure that valves and quick-connect fittings are in good working order; repair or replace as necessary to eliminate any problems.
  - Recoat chipped surface-water samplers with epoxy paint or "plasti-coat." Such samplers must be recoated before use.



[1] Substitute with DIW if tapwater is not available or of poor quality.

[2] Remove and clean metal parts, as shown for metal equipment.

EXPLANATION

DIW	Distilled/deionized water
methanol	Current protocol includes methanol rinse for most types of equipment except that used with samples for organic-carbon analyses. However, this protocol is under review. SAFETY ALERT: Methanol is highly flammable; fumes can be hazardous to human health.
PBW	Pesticide-grade blank water
VBW	Volatiles- and pesticide-grade blank water

Figure 3-1. General sequence for cleaning equipment before sampling for inorganic and (or) organic analytes.

- Rinse equipment with DIW directly after use while equipment is still wet and before cleaning procedures are implemented.
- Place cleaned equipment in doubled storage bags.

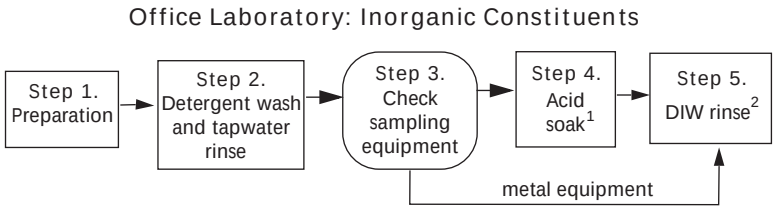
Do not allow collection and processing equipment to sit uncleaned in a field vehicle or elsewhere between field trips.

CLEANING OF EQUIPMENT USED TO SAMPLE FOR INORGANIC CONSTITUENTS 3.2.1

Cleaning of equipment used to collect and process water for analysis of inorganic constituents involves a five-step office-laboratory procedure or a five-step field-site procedure. These procedures are effective for cleaning equipment exposed to water containing concentrations of as much as 50,000 µg/L of iron, 5,000 µg/L each of manganese and zinc, 400 µg/L of copper, 125 µg/L of cobalt, and large concentrations of the other trace elements (Horowitz and others, 1994). The cleaning procedures are summarized in figures 3-2 and 3-3. (These procedures do not apply to field-measurement instruments—see NFM 6.)

Equipment should be cleaned periodically in the office laboratory, where complete disassembly is more practical and more thorough procedures are possible. Compared to cleaning at the field site, cleaning procedures carried out in the office laboratory involve longer exposure of equipment to cleaning solutions, more frequent change of cleaning solution, and greater volumes of rinse water.

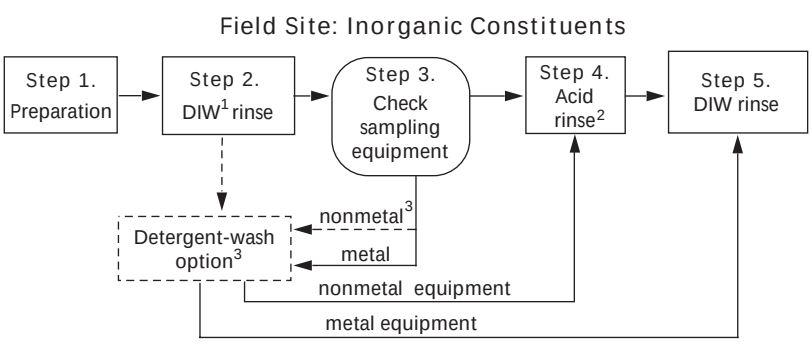
- To minimize field cleaning of equipment between sampling sites, preclean a separate set of equipment for each site.



[1] Acid soak used for nonmetal equipment.

[2] DIW = deionized water (table 3-1).

Figure 3-2. Office-laboratory cleaning procedures for equipment used to sample for inorganic constituents.



[1] DIW = deionized water (table 3-1).

[2] Acid rinse used for nonmetal equipment.

[3] Detergent wash is routine only for field cleaning of submersible pumps, metal equipment components, or excessively contaminated equipment.

Figure 3-3. Field-site cleaning procedures for equipment used to sample for inorganic constituents.

- If individual or dedicated sets of equipment for each field site are not available or cannot be precleaned, clean the equipment onsite and process additional field blanks during each field trip (Horowitz and others, 1994; Koterba and others, 1995).

- Return excessively contaminated equipment to the office laboratory for rigorous cleaning before reuse.

- After cleaning, document completion of and any modifications to the cleaning procedures.

Equipment-cleaning procedures for inorganic constituents

Standard procedures for office-laboratory and field-site cleaning of equipment used to collect and process samples for analysis of inorganic constituent are described below and summarized in figures 3-2 and 3-3. Not all the steps listed apply to all equipment, however. For example,

- Omit detergent step when cleaning plastic bags for surface-water samplers.

- Omit acid step when cleaning submersible pumps, the churn-splitter spigot, or other equipment constructed of stainless steel or other metallic material.

- Omit detergent and acid steps when cleaning sample bottles.

Be sure to check the specific procedures for sample bottles and other selected equipment listed in section 3.3 before proceeding with the office-laboratory and field-site procedures.

Step 1. Preparation at the office laboratory or field site (figs. 3-2 and 3-3).

a. Prepare a contaminant-free space for cleaning and drying the cleaning supplies and sample-collection and sample-processing equipment.

   i. Gather the cleaning supplies, the equipment to be cleaned, and the plastic bags or other material with which to wrap the cleaned equipment. Check table 3-1 for the cleaning supplies needed.

   ii. Place clean plastic sheeting over the work surface.

   iii. Put on disposable, powderless gloves[2], a laboratory coat or apron, and safety glasses.

   iv. Prepare the detergent solution, using a nonphosphate, laboratory-grade detergent.

   - Office laboratory (fig. 3-2). Use 0.1- to 2-percent solution, volume-to-volume (v/v), using a higher concentration for dirtier equipment.
   - Field site (fig. 3-3). Use 0.1- to 0.2-percent solution, v/v.

   v. Prepare the acid solution, using a 5-percent v/v dilution of ACS trace-element-grade hydrochloric acid (HCl) in DIW.

   - Add the acid to the water, not water to acid (NFM 9).
   - If nitric acid (HNO_3^-) will be used, prepare a 10-percent solution (v/v) of ACS trace-element-grade acid in DIW.

   vi. Label each washbasin, standpipe, and wash bottle to indicate the solution it will contain. Use a black waterproof marker.

   vii. Unwrap the equipment to be cleaned and discard the storage bags. Change gloves.

b. Clean the items used to clean the equipment.

   i. Fill washbasins and (or) standpipes with the nonphosphate detergent solution. Put wash bottles, scrub brushes, and other small items used for cleaning into a washbasin. Soak for 30 minutes.

   ii. Scrub interior and exterior sides of basins and standpipes with soft scrub brushes. Fill wash bottles with a soapy solution and shake vigorously.

[2]Refers to laboratory gloves that are nonpowdered on the inside and intended for disposal after one use. Glove materials must be appropriate for the work to be carried out and the solutions and equipment to be contacted. For example, vinyl gloves are appropriate for most sampling activities but not when working with methanol or other organic solvents.

iii. Rinse all items thoroughly with tapwater to remove detergent residue. No detergent bubbles should appear when fresh tapwater is agitated in the basin, standpipe, or wash bottle.

iv. Rinse washbasins with DIW.

v. Pour 5-percent HCl (or 10 percent HNO_3^-) solution into washbasins, standpipes, and wash bottles. Soak for 30 minutes. Do not soak items with metal parts (exposed or hidden) in an acid solution.

vi. Discard used acid solution into a neutralization container containing a bottom layer of marble chips (Step 4d).

vii. Rinse washbasins, standpipes, and wash bottles with DIW. Dispose of DIW using directions in Step 4d.

c. Disassemble sample-collection and sample-processing equipment. Change gloves.

- Submersible pumps should be disassembled periodically for office cleaning, but they are not usually disassembled for field cleaning.
- Processing and preservation chamber frames should be cleaned periodically using office-laboratory cleaning procedures. Field cleaning is needed only if the cover is slipped over the frame instead of being clipped to the inside of the frame.

Step 2. Detergent wash and tapwater rinse—Office laboratory (fig. 3-2).

a. Place small equipment parts into washbasin labeled for detergent and fill with a 0.1- to 2-percent solution of nonphosphate laboratory detergent. The amount of detergent depends on the hardness of the tapwater and the degree to which the equipment is dirty or contaminated.

b. Soak equipment and tubing for 30 minutes: fill tubing with solution and keep submerged.

c. Scrub exterior and interior of equipment surfaces to the extent possible, using a firm sponge or soft brush to remove any adhering material such as oil and grease, sediment, algae, and chemical deposits. Pay particular attention to grooves and crevices, O-rings, nozzles, and other spaces where inorganic or organic materials might be trapped. Change gloves.

d. Rinse equipment thoroughly with warm tapwater to remove detergent residue. Equipment rinsing is completed when no soap bubbles appear after the rinse water is agitated. Change gloves.

Step 2. DIW rinse and detergent-wash option—Field site (fig. 3-3).

For the DIW rinse:

a. Rinse equipment and tubing with DIW. Pay particular attention to removing material from grooves and crevices, O-rings, nozzles, and places where materials might be trapped. Note that equipment should already have had one DIW rinse directly after contact with sample water and before the equipment had a chance to dry.

b. Change gloves. Proceed to field detergent-wash option only for metal equipment components or for equipment that has become excessively contaminated.

For the detergent-wash option:

A field detergent wash is used for between-site cleaning of submersible pumps, metal components of equipment, or for equipment that has become greasy or otherwise coated and requires detergent to remove foreign materials; specific instructions for submersible pumps are given in section 3.3.9.

a. Place small equipment, tubing, and parts into basin labeled "detergent" and fill with a 0.1- to 0.2-percent detergent solution. Soak for about 10 minutes, or keep equipment assembled and circulate the solution through pump tubing for 5 to 10 cycles.

b. Scrub equipment surfaces with a firm sponge or soft brush to remove any adhering material such as oil and grease, sediment, algae, or chemical deposits. Pay particular attention to grooves and crevices, O-rings, nozzles, and other places where materials might be trapped. Change gloves.

c. Rinse equipment thoroughly with tapwater to remove detergent residue. Use DIW if tapwater is unavailable or is suspected of having a quality so poor as to contaminate the equipment. If necessary, use a wash bottle filled with DIW or tapwater to rinse hard-to-reach places; pump tapwater through assembled equipment for five or more tubing volumes. Equipment rinsing is complete when no soap bubbles appear after agitating the rinse water. If nonmetal equipment has been detergent-washed, go to Step 4.

d. Place equipment into acid-solution washbasin. Change gloves.

Step 3. Check equipment—Office laboratory and field site (figs. 3-2 and 3-3).

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— Nonmetal equipment or equipment with removable metal parts: remove any metal parts and go to Step 4.

— Metal equipment components or excessively contaminated equipment: go to Step 2, detergent-wash option at the field site and then to Step 5, DIW rinse.

Step 4. Acid soak/rinse—Office laboratory and field site (figs. 3-2 and 3-3).

For equipment constructed primarily of glass or fluorocarbon polymer or some other plastic, soak (office laboratory) or rinse (field site) in a 5-percent (v/v) HCl solution to remove any remaining organic films and inorganic deposits.

TECHNICAL NOTE: A 10-percent (v/v) HNO_3^- solution can be used instead of HCl if samples to be collected with the equipment will not be analyzed for nitrogen species.

CAUTION: Wear safety glasses and other protective apparel when working with acids.

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a. Place nonmetal equipment and tubing into the washbasin labeled "acid solution."

b. Office laboratory. Fill basin with dilute HCl solution (see TECHNICAL NOTE above). Soak equipment and tubing for 30 minutes. Carefully swirl the acid solution several times during the 30-minute soak to enhance removal of mineral encrustations.

c. Field site. Using a wash bottle filled with 5-percent HCl solution (see TECHNICAL NOTE above), rinse exterior of equipment and tubing. Pump acid solution through the equipment and tubing, using a peristaltic pump.

d. Carefully pour or pump the used acid solution into a neutralization container with marble chips covering the bottom (table 3-1). Do not reuse the acid solution.

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- Do not fill the neutralization container more than three-fourths full of acid solution.
- Ventilate container and workspace to allow for safe escape of carbon dioxide gas during dissolution of marble chips.

- Check the solution pH periodically using narrow range pH indicator strips. Neutralization is complete when the solution pH is greater than 6.0 or the original DIW pH.
- Discard the neutral solution, as appropriate.
- Rinse the container with tapwater but retain any undissolved marble chips. Replenish chips to form a layer on the bottom of the neutralization container.

Step 5. DIW rinse—Office laboratory or field site (figs. 3-2 and 3-3).

a. Place equipment into the cleaned washbasin labeled DIW. Change gloves.

b. Office laboratory. Rinse exterior and interior of each piece of equipment and tubing thoroughly with DIW and place on a clean surface to dry or into a clean IBW washbasin if blank samples will be collected to quality control the cleaning procedures.

c. Field site. Pump DIW through equipment.

d. Pour or discharge DIW rinse water into neutralization container. Change gloves.

e. Continue DIW rinsing until rinse-water pH is greater than 6.0 or the original DIW pH.

f. Allow equipment to air dry in an area free from potential airborne contaminants.

Storage of clean equipment

- Place dry, clean equipment inside doubled plastic bags. For small equipment, parts, and tubing, use sealable plastic bags.
- Place the churn splitter and funnel into doubled plastic bags and then place churn splitter inside of the churn carrier.

Clean equipment at the sampling site while equipment is still wet and before leaving for the next site.

CLEANING OF EQUIPMENT USED TO SAMPLE FOR ORGANIC COMPOUNDS 3.2.2

Nearly identical procedures are used in the office laboratory and at the field site to clean equipment used to sample for organic compounds. The office laboratory provides an environment in which equipment can be cleaned over an extended time using greater volumes of cleaning and rinsing solutions than in the field. The five-step cleaning procedure summarized in figure 3-4 is described in this section. If inorganic constituents also will be sampled for, check the sequence of cleaning solution to be used as shown in figure 3-1 before proceeding.

- Preclean a separate set of equipment for each site in order to avoid field cleaning of equipment between sampling sites. Always rinse equipment with DIW directly after use, however.
- If individual or dedicated sets of equipment for each field site are not available or cannot be precleaned, field clean equipment before moving to the next sampling site and process additional field blanks for each field trip (Koterba and others, 1995).
- Collect additional field blanks after cleaning equipment that was exposed to high levels of contamination (NFM 4) and before the equipment is reused for environmental sampling.

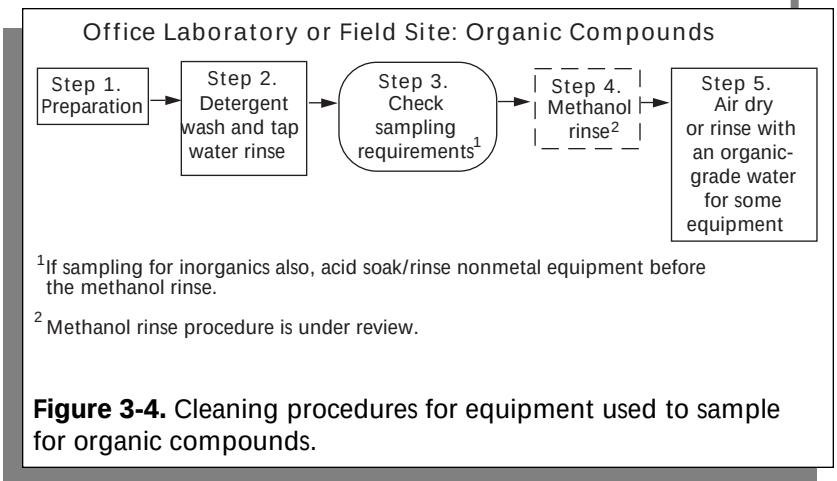

Figure 3-4. Cleaning procedures for equipment used to sample for organic compounds.

Equipment-cleaning procedure for organic compounds

Standard procedures for office-laboratory and field-site cleaning of equipment used to collect and process samples for organic-compound analysis are described below and summarized in figure 3-4. Not all the steps listed apply to all equipment, however. For example,

- Omit any cleaning procedure for sample bottles for organic compounds. Bottles for organic analyses arrive from the laboratory capped and ready for use and should not be rinsed by field personnel. Discard bottles if received uncapped.
- Omit the methanol rinse when cleaning the equipment used to collect and process samples for total, dissolved, and suspended organic carbon (TOC, DOC, SOC). If equipment (such as a submersible pump) that has been in contact with methanol or other organic solvent must be used for TOC, DOC, or SOC sampling, flush the equipment with copious quantities of sample water before collecting the sample; collection of a blank sample for DOC quality control is recommended.

Be sure to check the specific procedures for selected equipment listed in section 3.3 before proceeding with the office-laboratory and field-site procedures.

Step 1. Preparation (fig. 3-4).

a. Prepare a contaminant-free space for cleaning and drying the cleaning supplies and sample-collection and sample-processing equipment.

   i. Gather the cleaning supplies, the equipment to be cleaned, and clean storage bags and aluminum foil with which to wrap the cleaned equipment. (Check table 3-1 for the cleaning supplies needed.)

   ii. Cover the cleaning area with aluminum foil or fluorocarbon polymer sheeting.

iii. Put on disposable, powderless gloves,[3] a laboratory coat or apron, and safety glasses. Gloves provide protection from direct contact with solvents only for a limited period of time.

iv. Prepare the detergent solution, using nonphosphate laboratory-grade detergent. A 0.1- to 0.2-percent (v/v) solution is normally of sufficient strength, unless equipment is very oily or greasy. Do not use greater than a 0.2-percent solution for field cleaning.

b. Clean the items used to clean the equipment.

i. Label each washbasin, standpipe, and wash bottle with a black waterproof marker to indicate the solution it will contain.

ii. Follow Steps 2–5, listed below, to clean the washbasins, standpipes, wash bottles, and other items to be used for equipment cleaning.

c. Disassemble sample-collection and sample-processing equipment. Submersible pumps should be disassembled periodically for office cleaning but usually are not disassembled for field cleaning.

[3]Refers to laboratory gloves that are nonpowdered on the inside and intended for disposal after one use. Glove materials must be appropriate for the work to be carried out and the solutions and equipment to be contacted. For example, vinyl gloves are appropriate for most sampling activities but not when working with methanol or other organic solvents. Use solvent-resistant gloves when cleaning with organic solvents. Latex or nitrile disposable, powderless gloves are appropriate when using methanol.

Step 2. Detergent wash and tapwater rinse (fig. 3-4).

a. Place small equipment parts into washbasin labeled for detergent. Fill washbasin with a 0.2-percent solution of nonphosphate, laboratory-grade detergent. (The specific concentration of detergent solution depends on how contaminated the equipment might be and on the hardness of the tapwater.) Change gloves.

   - Office laboratory. Soak equipment in detergent solution for 10 to 30 minutes.
   - Field site. Rinse equipment exterior and interior with detergent solution.

b. Scrub the exterior and interior of equipment surfaces to the extent possible, using a firm sponge or soft brush to remove any adhering material such as oil and grease, sediment, algae, or chemical deposits. Pay particular attention to removing material from areas where inorganic or organic materials might be trapped, such as grooves and crevices, O-rings, and nozzles.

c. Place equipment into tapwater washbasin.

d. Rinse equipment thoroughly with tapwater to remove detergent residue. Use an organic-grade water (PBW, VBW, or office-produced) if tapwater is unavailable or is of a quality so poor as to contaminate the equipment. If necessary, use a wash bottle filled with organic-grade water or tapwater to rinse hard-to-reach places. Equipment rinsing is complete if no detergent bubbles appear when rinse water is agitated. Change gloves.

Step 3. Check sampling requirements (fig. 3-4).

a. If samples will be collected for organic analysis only, go to Step 4.

b. If samples will be collected for inorganic analysis in addition to organic analysis, follow the procedure for the acid wash and DIW rinse before proceeding with the methanol rinse (see figs. 3-1 and 3-4).

Step 4. Methanol rinse[4] (fig. 3-4).

a. Change to gloves that are chemically resistant to any solvent being used. Place cleaned equipment into a clean stainless steel or organic-solvent-resistant washbasin. Methanol-rinse area must be outside of the field vehicle and away from the sample-processing site. Sample-collection, -processing, and -preservation areas must remain free of solvent vapors.

CAUTION: Use methanol or other organic solvents sparingly and work under a fume hood or in a well-ventilated area, away from where an open flame or sparks can occur. Wear safety gloves, glasses, and apron.

b. Use pesticide-grade methanol (or appropriate organic solvent) dispensed from a methanol fluorocarbon-polymer wash bottle (office laboratory) or pumped through tubing (field site) (see TECHNICAL NOTE below).

i. Rinse equipment exterior and interior with a minimum amount of methanol.

ii. Rinse interior of pump tubing with methanol.

- Do not rinse exterior of pump tubing with methanol.
- Do not rinse pump tubing with methanol or any organic solvent if TOC, DOC, or SOC samples will be withdrawn through that tubing.

[4]Current (1998) cleaning protocol dictates the use of methanol to remove contaminants from equipment to be used to collect samples for analysis of organic compounds. This protocol is under review.

iii. Place equipment components and tubing on a clean aluminum foil surface.

iv. Pour or discharge used methanol (or other organic solvent) into an appropriate waste container for flammable liquids (Water Resources Division Memorandum 94.007). Change gloves. Dispose of gloves used for methanol rinse appropriately.

TECHNICAL NOTE: Rinse with dichloromethane or hexane if the methanol rinse is not sufficient to clean equipment contaminated with excessive concentrations of hydrophobic organic compounds. If rinsing with dichloromethane or hexane, use pesticide-grade solutions, wear nitrile gloves, and use only on dry equipment (dichloromethane and hexane are not soluble in water). Do not rinse equipment with any organic solvent if equipment will be used for TOC, DOC, or SOC samples.

Step 5. Air dry equipment or rinse with organic-grade water (fig. 3-4).

a. Allow methanol-rinsed equipment to air dry in an area free from dust and potential airborne contaminants (place an aluminum foil tent loosely over the drying equipment).

b. If it is not practical for the methanol to evaporate from the interior of equipment components or sample tubing, either

- dry by blowing clean, filtered, inert gas through equipment; or
- rinse methanol from equipment with pesticide-grade or volatile-grade blank water, dispensed from a wash bottle or pumped with a valveless fluid metering pump.

Storage of clean equipment

Cover all equipment orifices with aluminum foil or fluorocarbon polymer bags, then place equipment into sealable storage bags. Isolate equipment used to collect trace-element samples from aluminum foil.